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Ionic Doping of Low-Conductivity Structural Resins for Improved Direct-Current Sensing

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Abstract

This investigation developed a methodology for doping high-resistivity vinyl-ester (VE) resins with an organic dopant. The polymeric resin system investigated was a Dow Derakane 411-C50 VE resin. A number of potential dopants were studied, and two in particular, tetrabutylammonium acetate (TA) and tetrabutylammonium iodide, were found to be capable of increasing the ionic conductivity of VE resin without adversely affecting the resin viscosity, mechanical properties, or reaction kinetics. The primary candidate dopant that was characterized in this investigation was a TA organic salt. TA, at a 0.1-weight-percent (wt%) concentration, was shown to have negligible effects on the mechanical properties and reaction kinetics of a curing VE part. TA was also found to slightly increase the rate at which viscosity increases, but not to the extent that would hinder resin transfer molding of a doped VE system. This investigation has proven that doping of VE resin with 0.1-wt% TA is a viable means of controlling and tailoring the conductivity of high-resistivity resins for the application of direct-current (DC)-sensing technology.

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1. Introduction

As the composites industry grows, there is an increasing need to improve quality and reduce the cost of producing composite structures. To meet these demands, research has focused on the development and refinement of new technologies to sense the condition of a composite during manufacturing. SMARTweave is a patented sensor system that measures and tracks the direct-current (DC) conductance of a resin as it flows into a preform and cures [1–4]. This is done through an overlapping, but not touching, grid of wires; one set serves as excitation leads, and an orthogonal set in a parallel plane serves as sensing leads. The resin acts as a resistor. Under an applied voltage, current is induced in a circuit that enables the conductance to be monitored during the process.

One common approach in single-point flow and cure sensing is to monitor the dielectric response of a material by the application of an alternating current. However, such techniques track not only the ionic conductivity of the curing resin but a more complex dielectric response. The use of a DC measurement technique allows for simpler measurement of DC conductance and for rapid continuous multiplexing of potentially thousands of "point sensors" in a mold.

Potential difficulties are associated with DC sensing of low-conductivity resin systems. Vinyl-ester (VE) resin systems are widely used in liquid-molding processes because of their low viscosity and ability to cure at room temperature. Low-viscosity room-temperature-cure resin systems offer reduced production costs and are therefore widely used by many composites manufacturers. However, the low conductivity of VE systems makes it difficult for a DC-sensing system to sense the presence and cure of the resin.

This paper investigates dopants that will raise the conductivity of high-resistivity resin systems. The effects of resin chemistry and doping on ionic conductivity are characterized through the use of SMARTweave single-node test cells. Further, the effects of doping on the mechanical properties, viscosity, and reaction kinetics of a curing VE resin are investigated

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through mechanical testing, viscometry, and transmission Fourier-transform infrared (T-FTIR) spectroscopy.

2. Background and Theory

Two basic groups of parameters govern the operation of DC-sensing systems: (1) the geometric configuration of the sensing system and (2) the material properties of the resin system. The geometric parameters of interest are conductor separation distance and surface area. The conductor separation distance influences the time it will take mobile charge carriers, ions, to migrate across the potential difference created between the conductor surfaces. The conductor surface area affects the number of ions that transfer charge from the liquid medium to the conductor in a unit time. The material properties of interest are viscosity, concentration of ions, and size of ions. Both the resin viscosity and dopant ionic radius influence the mobility of the ions.

This investigation is concerned with the effects of the material parameters and doping on the ionic conductivity of VE resin. The resin system under investigation is the Dow Derakane VE 411-C-50 resin system with 50% styrene (ST) content, designated VE. Figure 1 shows the chemical structure and makeup of VE and ST monomers. Cobalt napthenate (CoNap) is used as an accelerator along with a cumene hydroperoxide-based catalyst, Trigonox 239A. The system is mixed with 2.0-weight-percent (wt%) Trigonox 239A and 0.2-wt% CoNap then cured at room temperature. A number of potential charge carriers were investigated for their ability to lower the resistivity of the VE resin system by increasing the concentration of ions.

When choosing a dopant for the VE system, care was taken to choose a dopant that would ionize and separate into its ionic components, but not adversely affect the curing resin. An organic salt with a weak cation/anion pair is needed in order to avoid cross-linking of the salt with the vinyl groups, called a complex. Acceptable dopants would also have a short ionization time and low cost, relative to the other resin components. Through doping with a binary

Methacrylated DGEBA Monomer - Vinyl Ester
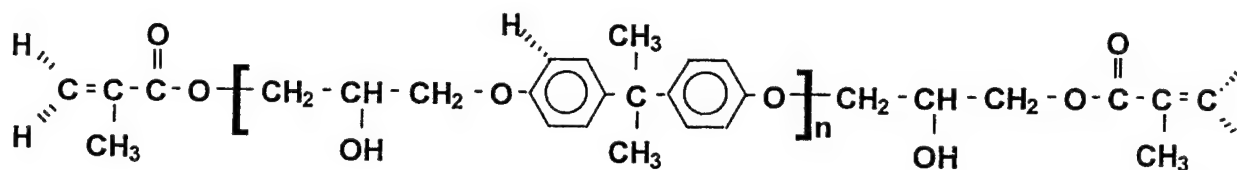
Styrene
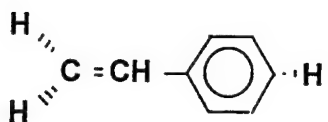
Figure 1. VE and ST Monomer Structures and Chemical Makeup.

compound, the sizes and interactions between ions could be determined. The resistivity of the resin in this case is a function not of resin chemistry, but of dopant concentration. In this way, the resistance of doped VE resin could be investigated and the relationship between ion concentration and DC sensing determined.

In work by Schwab, Levy, and Glover [5], the resistance of a parallel plate DC-sensing system was reported to be a function of the separation distance between plates (L), the surface area of the plates (A), the viscosity of the resin (η), the charge of ions in the system (Q_i), the concentration of the ions in the system (C_i), and the radius of the ions in the system (r_i), or

$$PR = \frac{6\pi\eta L\pi\eta\Lambda}{AA\sum_i \frac{C_iQ_i^2}{r_i}} , \qquad (1)$$

where viscosity is in Pascal-seconds, length in meters, area in square meters, concentration in numbers of ions per cubic meter, charge in coulombs, and ionic radius in meters. This relationship was developed by balancing the viscous drag force acting against an ion as it moves

through a resin and the electromotive force driving an ions toward and away from the charged plates.

For the SMARTweave DC-sensing system, the geometric parameters of L and A are determined by the junction formed by the sense and excitation leads. Due to the dependence of resistance on the concentration, charge, and size of ions, doping offers an effective means of lowering the resistance of low-conductivity polymeric materials.

In polymeric materials, two modes of electrical conduction are possible: (1) ionic modes and (2) electronic modes. In the former case, current flow results from the motion of ions. The latter involves motion of electrons along a conjugated polymer chain. In a curing system, no system of conjugation exists; thus, one relies on polar/ionic charge carriers to increase ionic conductivity. Ionic conductivity in polymers has recently gained increased attention because of applications in energy-conversion devices and chemical sensors [6, 7]. Ionic conductivity is the product of three terms: (1) the carrier charge (Q_i in coulombs), (2) the concentration of charge carriers (C_i in ions per cubic meter), and (3) the average velocity of a charge carrier in an electric field (S in meters per second). Thus, one can write an expression for the specific conductivity as

$$\sigma = Q_i C_i S. \tag{2}$$

Most polymers are poor ionic conductors because they are primarily organic and the salts are insoluble in the polymer matrix. The applications of polymeric ionic conductors require a polar ion-solvating polymer like poly(ethylene oxide).

The addition of an organic salt to a nonpolar resin does not guarantee an increase in ionic conductivity. A salt with a high degree of ionization in the polymer matrix with a small effective hydrodynamic volume of the resulting ions will result in maximum conductivity. The degree of ionization depends on the strength of association between counterions, water hydration, and complex formation. The strength of association for a simple ionic bond is given by Coulomb's law as

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$$E = \frac{Q^2}{4\pi\varepsilon_0 \varepsilon d}, \tag{3}$$

where Q is charge ($1.602*10^{-19}$ C), ε_0 is the permittivity of free space ($8.85*10^{-12}$ F/m), ε is the dielectric constant of the medium (3.32 for VE resin), and d is the distance between ionic cores in meters. Equation (3) applies well when hydration energies and metallic bonds are not important. It suggests that increased ionic radii will increase the degree of ionization in low dielectric materials.

From the limiting ionic conductivity of each ion, a hydrodynamic radii is estimated [8]. The limiting ionic conductivity for these calculations was determined in water; thus, they include the effective size of the hydrated ions. The resin system used in this study does contain trace amounts of water, and hydrated ions are expected to be present.

The degree of ionization, the equilibrium constant, and the concentration of ions can be calculated using equations (4) through (8). The measured molar ionic conductivity (Λ) is calculated as

$$\Lambda = \frac{1}{\rho_v C_d}, \tag{4}$$

where C_d is the concentration of dopant in grams, and ρ_v is the resistivity of the resin in ohms-meter. The resistance of the resin system being used can be measured using a DC conductivity cell. Once resistance has been measured with a DC cell, resistivity can be calculated by the following equation:

$$\rho_v = \frac{2\pi L_c R}{\ln \frac{D_2}{D_i}}, \tag{5}$$

where R is the measured resistance of the material in ohms, L_c is the length of cylinders in meters, D_1 is the outer diameter of the inner cylinder in meters, and D_2 is the inner diameter of the outer cylinder in meters.

The degree of ionization (ψ), and equilibrium constant (k) can be calculated using equations (6) and (7):

$$\Psi = \frac{\Lambda}{\Lambda_{max}}, \tag{6}$$

and

$$k = \left[\frac{\Psi^2}{1-\Psi}\right]C_d. \tag{7}$$

The concentration of ions (C_i) in solution within the resin system can be calculated as

$$C_i = \frac{\Psi M}{M_w V}6.023*10^{23}, \tag{8}$$

where M_w is the formula weight of the dopant used in grams per mole, M is the mass of dopant in grams, and V is the volume of the resin-dopant mixture in cubic centimeters.

The hydrodynamic radius of tetrabutylammonium and acetate ions can be calculated using equations (2) through (7) and the concept of limiting ionic conductance. The hydrodynamic radius of tetrabutylammonium and acetate ions was calculated to be 5.5 and 3.75 Å, respectively.

3. Experimental Procedures

In the following sections, the basic components and operation of a SMARTweave DC-sensing system are reviewed. The experimental apparatus and procedures designed to

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investigate the effects of resin chemistry on ionic conductivity are provided. In the last section is a description of the down-selection methodology used to determine the primary candidate dopant.

3.1 SMARTweave System Components and Operation. SMARTweave is a patented [1] sensor system that measures and tracks the DC conductance of a resin as it flows into an RTM mold and cures. This is done through a noncontacting grid of wires; one set serves as excitation leads and an orthogonal set in a parallel plane serves as sensing leads. Figure 2 shows a circuit diagram for a single-node voltage experiment. The resin acts as a resistor designated as R_j. Under an applied voltage, current is induced in a circuit containing a second resistor (R_s) of known resistance. The sense resistor, R_s, has a fixed resistance, which is the same order of magnitude of the resin resistance (R_j).
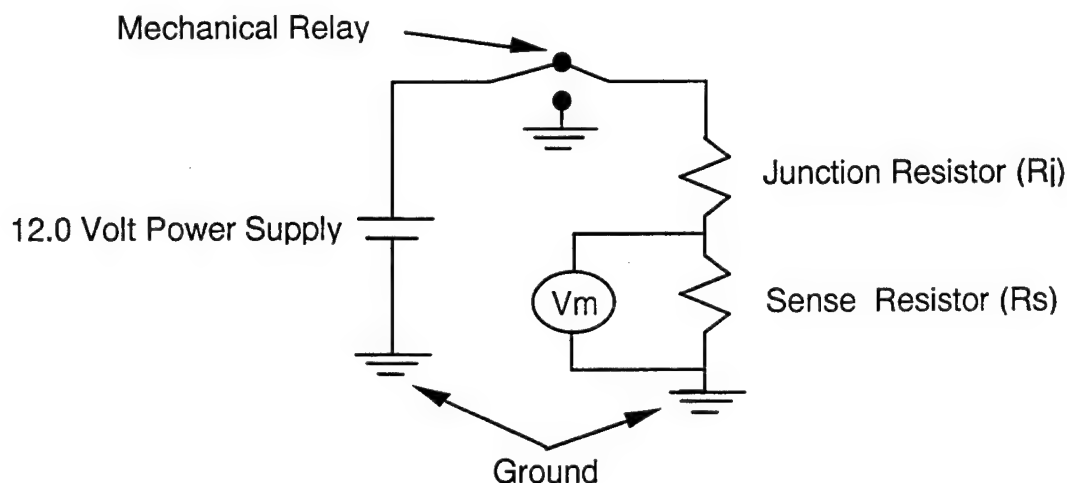
Figure 2. SMARTweave Single-Node Circuit Schematic.

The SMARTweave system used in this study consists of a National Instruments SCXI interface chassis (where the excitation and sense leads are connected), a National Instruments multiplexer (for switching from one set of leads to another), and a computer (for data display and storage). A 12-V power supply provides a DC voltage to the excitation leads. Only one excitation lead is active at any time. The multiplexer sends out a voltage to the excitation leads,

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and the SCXI chassis monitors the sense leads for an induced current. The induced current is detected as a voltage across R_s.

During a single-node test, a flat 12-V excitation voltage remains across a single excitation lead. In the presence of a conductive medium, such as a VE resin, a current is induced in the sense lead and a voltage is created across R_s. Changes in $R_j(t)$ are then recorded as changes in $V_m(t)$ by the SMARTweave Labview software.

For the single-node experiments performed in this study, a 12-V applied voltage and a 10-$M\Omega$ sense resistor were used. The measured voltage, $V_m(t)$, and sense resistance can be used to calculate the junction resistance, $R_j(t)$, by

$$R_j(t) = \left(\frac{12\,V}{V_m(t)}\right) \cdot R_s - R_s, \tag{9}$$

where $V_m(t)$ is in volts and R_s is in ohms.

Figure 3 is the schematic of a single-node test cell (SNTC). A SNTC consists of a sense and excitation lead that are made of stainless steel wire and attached to a box frame made of fiber-reinforced PVC. SNTC apparatus were designed to hold the geometric parameters, L and A, constant so that the material parameters could be studied.

3.2 Effects of Resin Chemistry on Ionic Conductance. SMARTweave experiments using SNTC apparatus were performed to study the effects of CoNap and Trigonox 239A on the conductance of VE resin. VE 411-C-50 resin samples were prepared by first adding the desired concentration of CoNap, then adding the desired concentration of Trigonox 239A, and mixing thoroughly. Once the accelerator was added into the VE-CoNap mixture, the specimen was poured into a SNTC and the SMARTweave voltage was monitored.

In the first series of experiments, the weight-percentage concentration of CoNap was varied from 0.22 wt% to 0.35 wt%, with a total resin mass of 50.0 g. The weight-percentage

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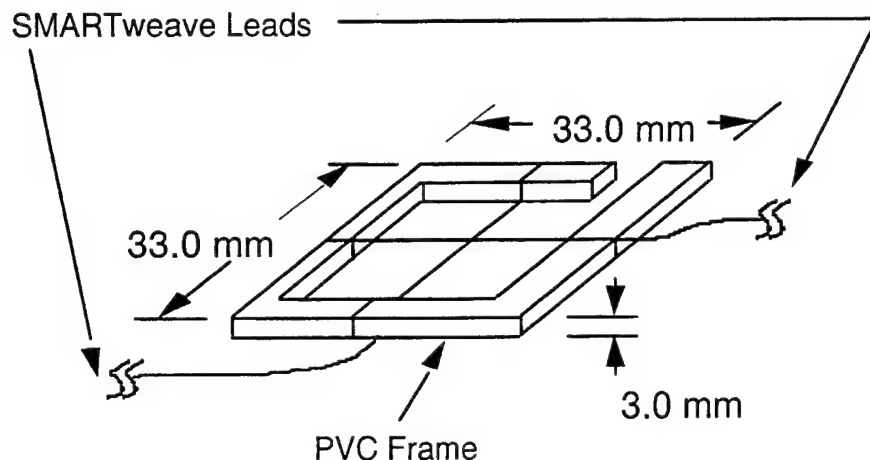
Figure 3. SNTC.

concentration of Trigonox 239A was held constant at approximately 2.0 wt%. In the second series of experiments, the weight-percentage concentration of Trigonox 239A was varied from 2.0 wt% to 3.5 wt%, with a total resin mass of 50.0 g. The weight-percentage concentration of CoNap was held constant at approximately 0.2 wt%.

3.3 Dopant Down-Selection and Characterization. The following section is broken into two parts: (1) dopant down-selection and (2) dopant-resin characterization. The first section describes the methodology used to determine the primary candidate dopant. The second section describes the experimental apparatus and procedures for the characterization of (1) the effects of doping on the viscosity of VE resin and (2) the effects of doping on the reaction kinetics of VE resin.

3.3.1 Dopant Down-Selection. The first priority of this investigation was to determine a suitable dopant for VE resin. Sodium acetate, sodium bisulfate, sodium salt, tetrabutylammonium acetate (TA), tetrabutylammonium hydroxide (TH), and tetrabutylammonium iodide (TI) were added to the VE resin system, and the voltage profile during cure was measured. A SNTC configuration was used to measure the SMARTweave voltage vs. time response of the curing candidate dopant and VE resin mixtures.

The effects of doping VE with the primary candidate dopant were then studied by varying the weight-percentage concentration of dopant from 0.025 wt% to 0.1 wt% with a total resin mass of 50.0 g. The weight-percentage concentrations of CoNap and Trigonox 239A were held constant at 0.2 wt% and 2.0 wt%, respectively.

Two panels, one with dopant added and one without, were manufactured using Seemann Composite Resin Infusing Molding Process (SCRIMP) manufacturing and monitored with SMARTweave process at room temperature. This was done to determine the effects of doping with the primary candidate dopant on the ionic conductivity and mechanical properties manufactured VE composite part. No post-curing was performed. Each part consisted of an identical lay-up of six layers of 5×4 24-oz E-glass fabric, cut into roughly 12-in squares. Stainless steel wire sensor leads were used to monitor the SMARTweave voltage of the curing part.

The nondoped resin plate consisted of 2.0-wt% Trigonox 239A, and 0.2-wt% CoNap in VE. The doped resin plate consisted of 0.1-wt% dopant, 2.0-wt% Trigonox 239A, and 0.2-wt% CoNap in VE. Short-beam shear and open-hole compression, A-695, specimens were cut from each panel and tested. Short-beam shear tests were conducted in accordance with American Society for Testing of Materials (ASTM) test standard D2344.

3.3.2 Characterization. The resistance of a doped VE resin sample was monitored over the period of 18 hr to determine the amount of time needed to reach the equilibrium concentration of ionized dopant. The sample consisted of 0.1-wt% dopant mixed, only partially, with VE; no initiator or accelerator was added.

The resistance of VE doped with 0.05–4.8-wt% dopant was measured using a cocylindrical DC conductivity cell and a Fluke multimeter. The DC conductivity cell design was constructed by following the ASTM -test standard D257-78. Samples with the aforementioned concentrations were then prepared and shelved until an equilibrium concentration of ions was reached. A DC cell was then filled with one sample at a time and used to measure the resistivity

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of the resin. The measured resistivity was then used along with equations (3) through (7) to determine the ionic concentrations. Figure 4 shows the shape and dimensions of the DC cell used in this experiment.
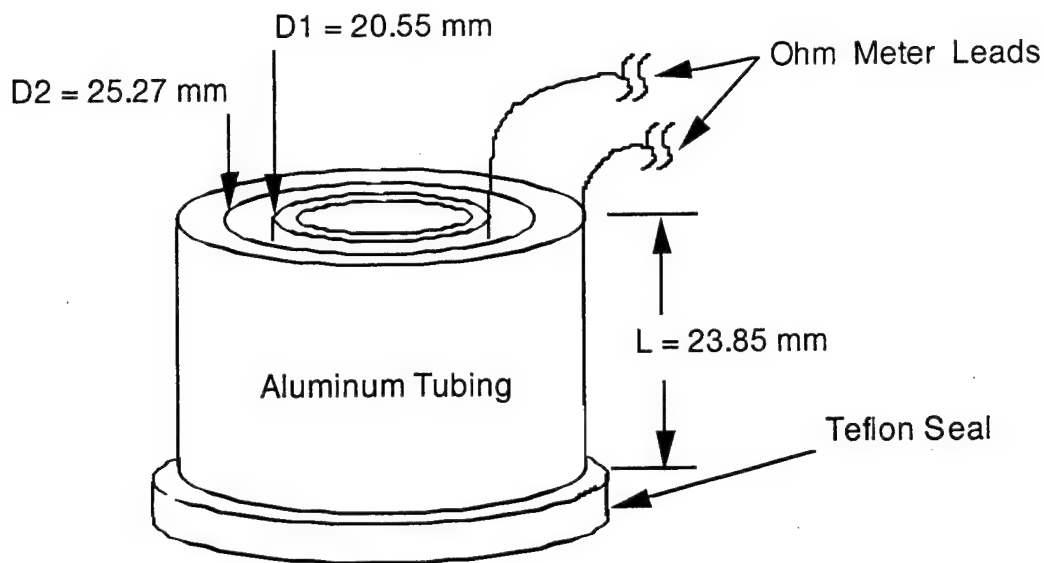
Figure 4. DC Cell Configuration.

The viscosity of both a nondoped and doped curing VE resin was measured using a Brookfield Viscometer and Small Sample Adapter, which was capable of regulating temperature via water jacket and circulating water bath. The temperature was held constant at 30 °C. The doped VE resin system consisted of 0.1-wt% dopant, 2.0-wt% CoNap, and 0.2-wt% Trigonox 239A.

T-FTIR spectroscopy is a characterization method that can be used to determine the degree of conversion of VE and ST monomers of a curing VE resin system [9]. By measuring the degree of conversion of the individual monomers, the effects of doping, if any, on the reaction kinetics of VE resin can be determined.

Degree of conversion experiments were performed on a Nicolet 20DXB Fourier transform infrared (FTIR) spectrometer in transmission mode. T-FTIR experiments were performed at 30, 40, and 50 °C. Specimen temperature was controlled by a set of heater cartridges which are embedded within the specimen holder.

4. Results and Discussion

The following section reports the effects of resin chemistry on the ionic conductivity of VE resin. The purpose of these experiments was to characterize the sensitivity of ionic conductivity to variations in resin chemistry and to determine the viability of using different component concentration to enable DC sensing. For the recommended concentrations of initiator, Trigonox 239A at 2.0 wt%, and accelerator, CoNap at 0.2 wt%, the initial SMARTweave voltage is 0.6 V with a 60-min duration of measurable conductivity.

Through doping, the initial SMARTweave voltage can be increased to over 2.5 V. The duration of measurable ionic conductivity remains unaffected; however, the sight-to-noise (S/N) ratio is greatly increased, which increases the resolution of DC sensing and enables the in-situ determination of gelation and possible cure to greater extents of reaction.

The effects of resin chemistry on ionic conductivity and the results of the dopant down-selection studies are presented. The latter sections report the effects of doping on the initial SMARTweave voltage, mechanical properties, viscosity, and reaction kinetics of VE resin.

4.1 Effects of Resin Constituents on Ionic Conductance. Figure 5 shows SMARTweave voltage vs. time for variable concentrations of CoNap. There is a slight increase in voltage with increasing weight-percentage concentrations of CoNap. The increased peak voltage is the result of faster reaction kinetics and higher exothermic temperatures that lead to lower resin viscosity and increased conductance.
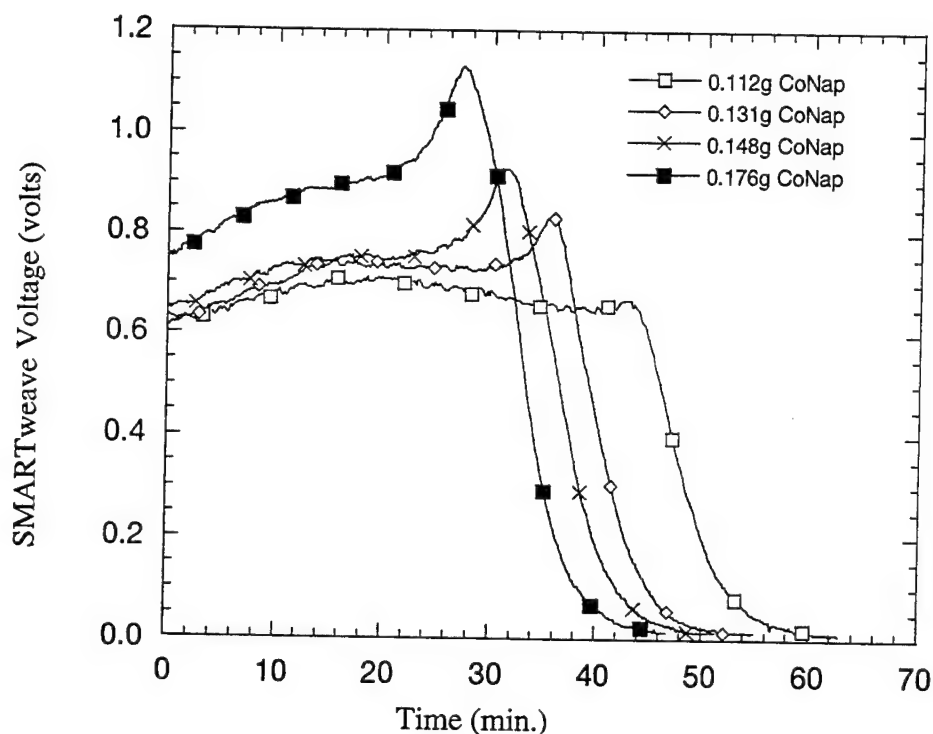
Figure 5. Effect of Variable Concentrations of CoNap on Ionic Conductance. Markers Do Not Represent All Data Points Taken. All Data Points Fall on Lines Connecting Markers.

For these experiments, the resin starts to cure the instant that all of the components have been mixed together. The increase in voltage up to the point at which the exotherm occurs is the result of heat released by the reaction. The reacting polymer system reacts as a polymer electrolyte up to the start of gelation. This can be seen in the rise of ionic conductivity with increasing heat of reaction.

Figure 6 shows the change in initial SMARTweave voltage ($V(t = 0)$) and the time at which voltage goes to zero ($t(V = 0)$) with increasing concentration of CoNap. The slight increase in $V(t = 0)$ with increasing concentrations of CoNap proves that CoNap adds only a small percentage of impurity ions to the resin system.
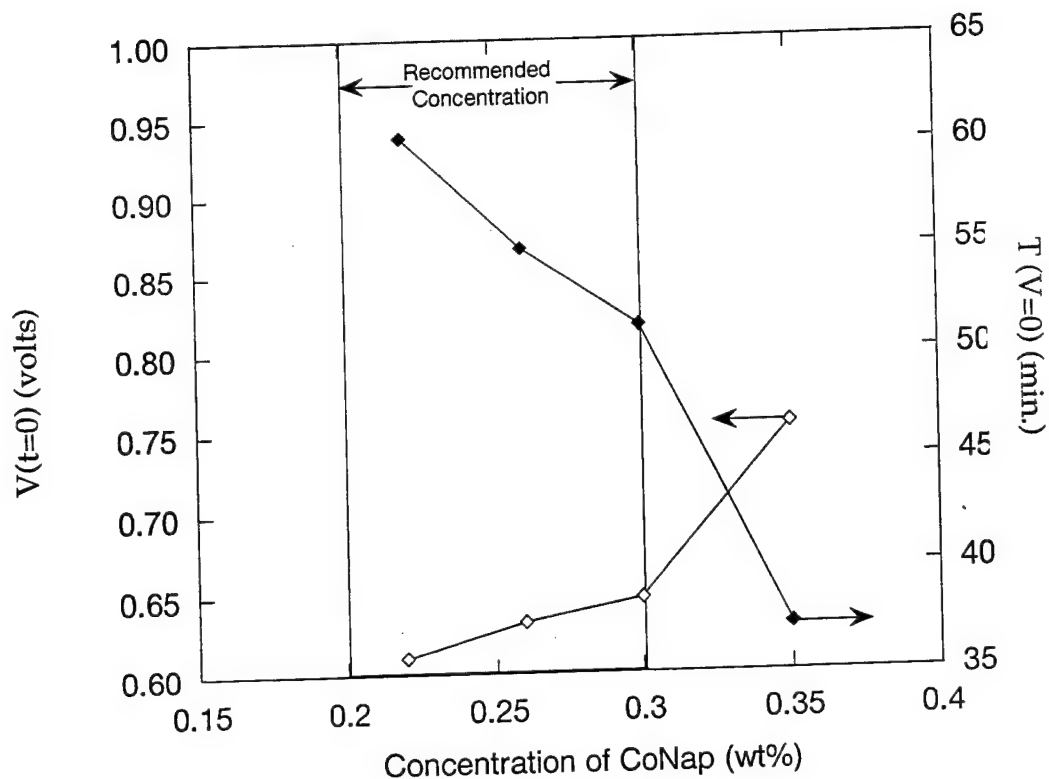
Figure 6. Initial SMARTweave Voltage vs. Concentration of CoNap. Data Points Are Connected by Lines.

In the recommended region of CoNap concentration, $V(t = 0)$ is a linear function of concentration. A linear fit to that regions yields a line with a slope of 0.46 V/wt%. The slight increase in initial voltage comes at the cost of over a 20-min decrease in period of measurably ionic conductivity. The decrease in $t(V = 0)$ is the result of accelerated reaction kinetics, which are dependent not only on the concentration of initiator (Trigonox 239A) but on the concentration of accelerator as well (CoNap). This makes the addition of more CoNap to the system an unacceptable way of increasing ionic conductivity.

Figure 7 shows the change in voltage as a function of time for variable concentrations of Trigonox 239A. There is a notable increase in initial voltage with increasing weight-percentage concentrations of Trigonox 239A. The increased peak voltage is the result of faster reaction
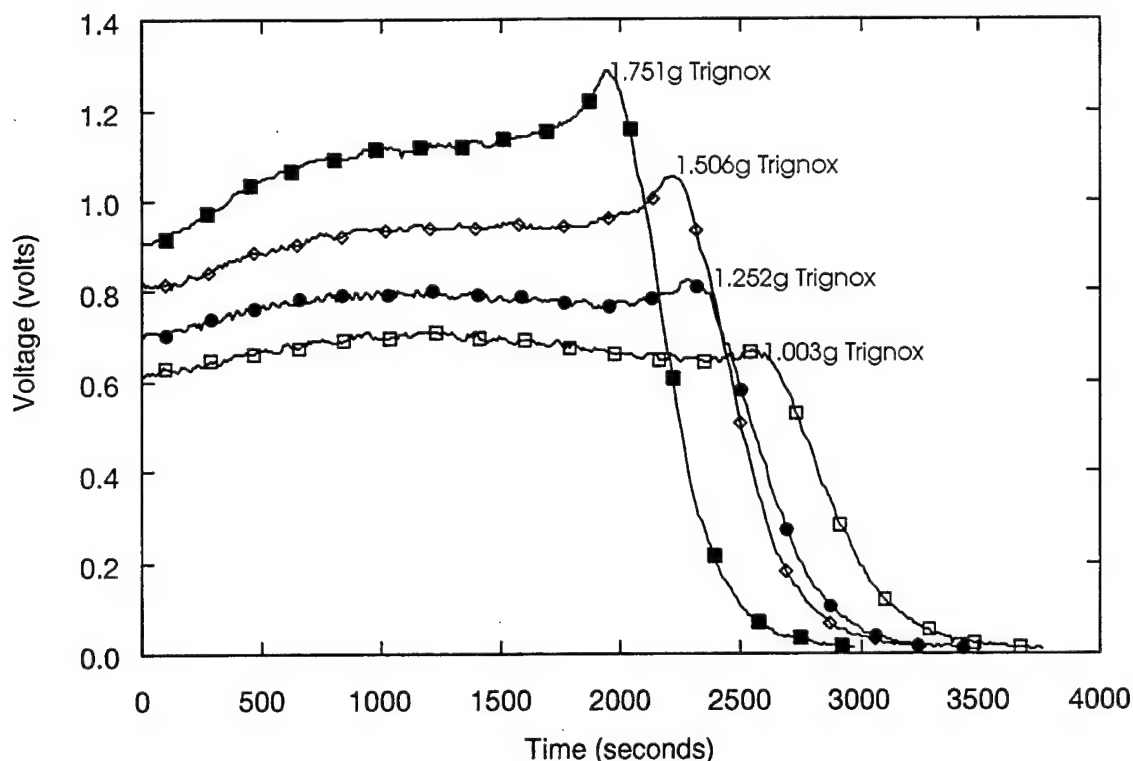
Figure 7. SMARTweave Voltage vs. Time for Variable Concentrations of Trigonox 239A. Markers Do Not Represent All Data Points Taken. All Data Points Fall on Lines Connecting Markers.

kinetics and higher exothermic temperatures that lead to lower resin viscosity and increased ionic conductance.

Figure 8 shows the change in initial voltage ($V(t = 0)$) and the time at which the voltage goes to zero ($t(V = 0)$) with increasing concentration of Trigonox 239A. The increase in $V(t = 0)$ with increasing concentrations of Trigonox 239A proves that the initiator adds a significant percentage of impurity ions to the resin system.

In the recommended region of Trigonox 239A concentration, $V(t = 0)$ is a linear function of concentration. A linear fit to that regions yields a line with a slope of 0.21 V/wt%. The increase in initial voltage, however, comes at the cost of a significant decrease in the period of

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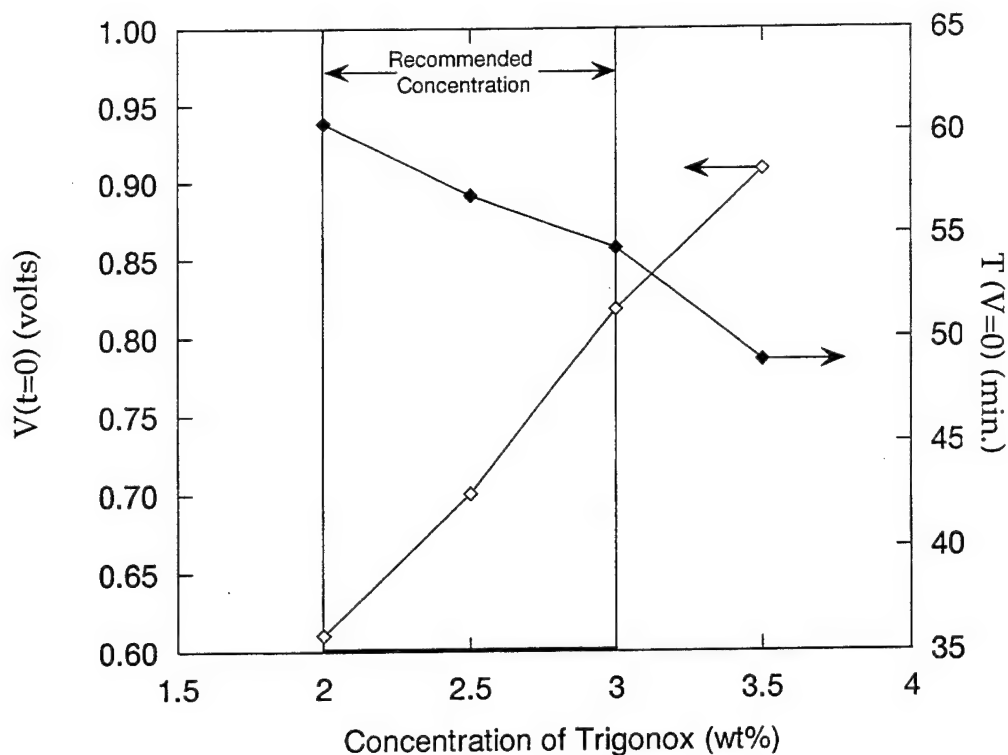
Figure 8. Initial SMARTweave Voltage vs. Concentration of Trigonox 239A. Data Points Are Connected by Lines. Line of Linear Curve Fit for Recommended Region Is Not Shown.

measurably ionic conductivity. The drastic decrease in $t(V = 0)$ makes the addition of more Trigonox 239A to the system an undesirable way of increasing ionic conductivity. The decrease in $t(V = 0)$ is the result of accelerated reaction kinetics, which are dependent on both the concentration of accelerator (CoNap) and on the concentration of initiator (Trigonox 239A).

From a processing perspective, accelerated cure kinetics and reduced time to gelation are not desirable. This is mainly due to the finite amount of time needed for resin infusion and the detrimental effects increased heats of reaction may have on the mechanical properties of a manufactured part.

4.2 Dopant Down-Selection and Characterization. The following sections provide the results of the dopant down-selection and characterization of the primary candidate dopant, including the effects of doping on ionic conductivity, viscosity, and reaction kinetics of VE resin.

4.2.1 Dopant Down-Selection. Figure 9 shows the effects of the various candidate dopants on the ionic conductivity on curing VE. Sodium acetate is not shown in Figure 9 because it caused immediate cross-linking of the VE system. Therefore, sodium acetate could not be monitored with a SMARTweave system. The sodium bisulfate and sodium salt additives showed a low solubility in the VE system, which explains the low increase in voltage signal. The tetrabutylammonium ion pairs all showed an ability to partially ionize in the VE system. The TH- and TI-doped VE systems showed a lower increase in conductivity than the TA-doped VE system. As a result of its ability to increase the conductivity of VE resin without any signs of adversely affecting cure, TA was chosen as the primary candidate dopant for continued investigation.
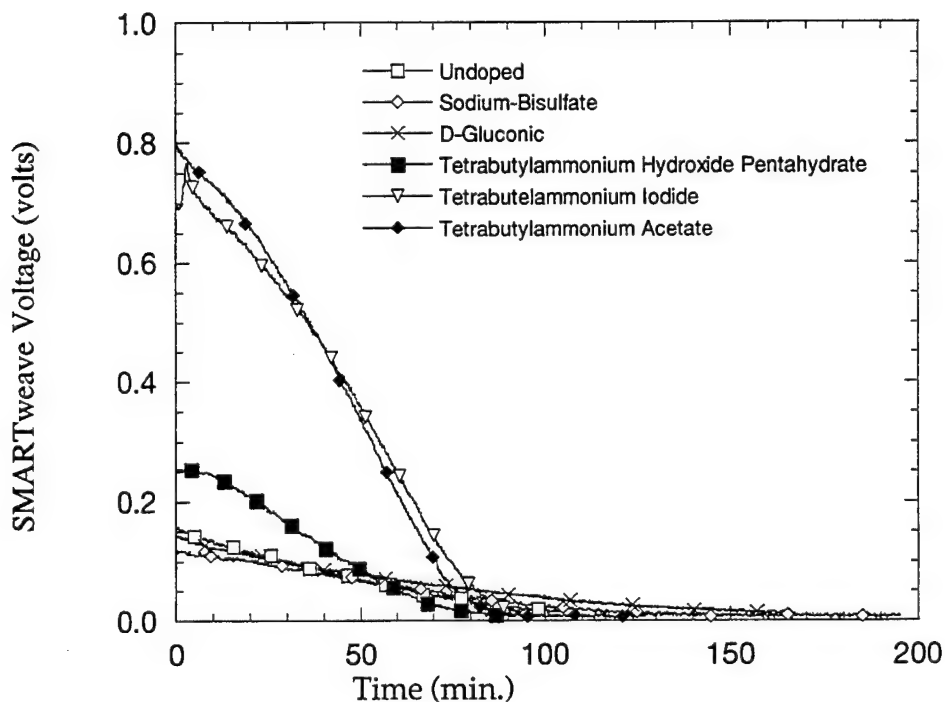
Figure 9. **SMARTweave Voltage vs. Time for Candidate Dopants. Markers Do Not Represent All Data Points Taken. All Data Points Fall on Lines Connecting Markers.**

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4.2.2 Characterization of TA-VE Resin System.

4.2.2.1 Effects of Doping on Ionic Conductivity. Figure 10 shows the effects of increased concentration of TA on ionic conductivity. This figure clearly shows that increasing the concentration of TA raises ionic conductivity, but does not adversely affect the SMARTweave voltage profile of the curing VE resin system. In other words, whereas increased concentrations of CoNap and Trigonox 239A caused exotherms and decreased periods of measurable voltage, TA showed no such effects.
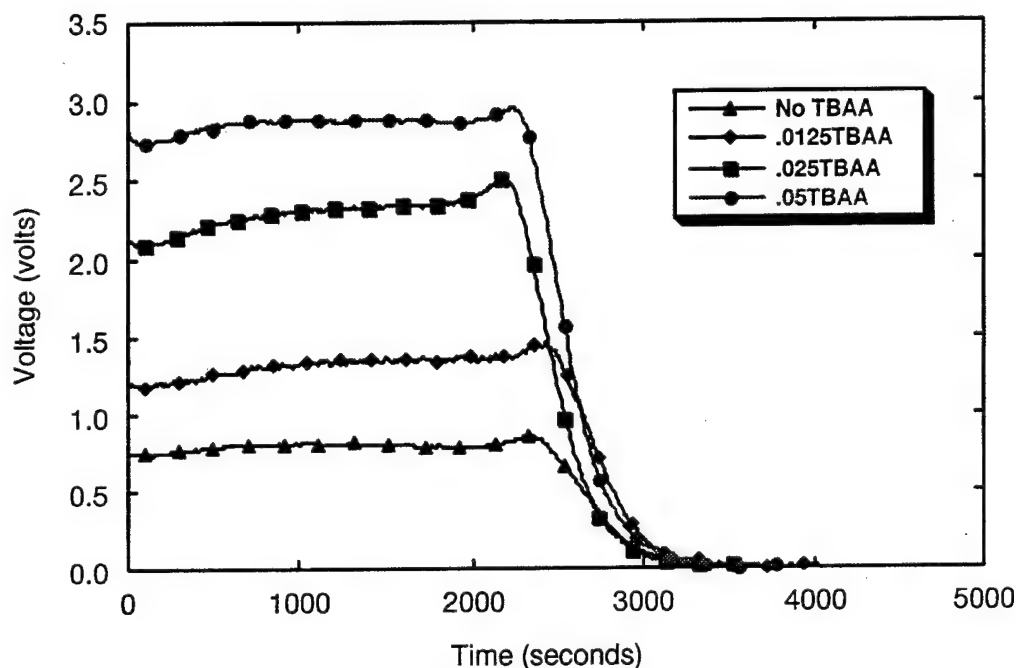
Figure 10. Effect of Variable Concentration of TA on SMARTweave Voltage. Markers Do Not Represent All Data Points Taken. All Data Points Fall on Lines Connecting Markers.

Figure 11 shows the change in initial SMARTweave voltage ($V(t = 0)$) and the time at which SMARTweave voltage goes to zero ($t(V = 0)$) as a function of weight-percent concentration of TA. The increase in $V(t = 0)$ with increasing concentrations of TA prove that TA adds a significant percentage of impurity ions to the resin system.

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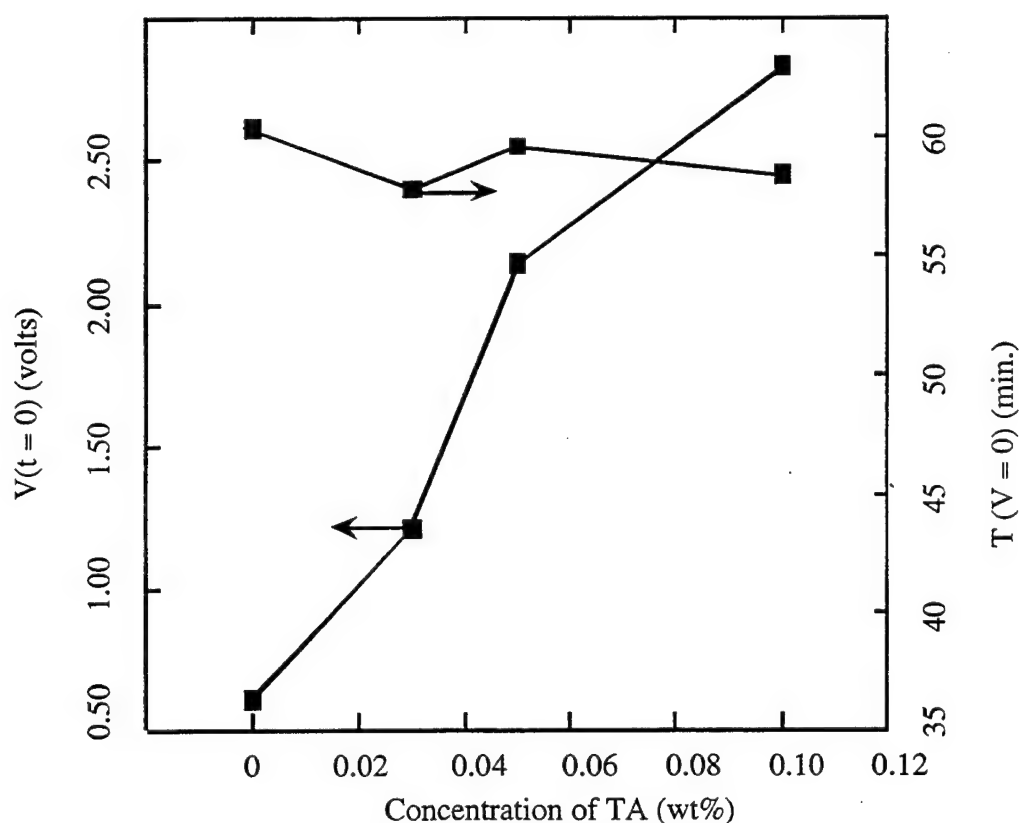
Figure 11. Initial SMARTweave Voltage vs. Concentration of TA. Data Points Are Connected by Lines.

For all TA concentrations tested, $V(t = 0)$ is a linear function of weight-percent concentration. A linear fit to those regions yields a line with a slope of 22.4 V/wt%. Note, that this is an order of magnitude higher than CoNap and Trigonox 239A. Ionic conductivity can now be adjusted to the level needed for muliplex sensing by simply adding more dopant.

The increase in initial voltage comes with no significant change in the period of measurably ionic conductivity. The ability of TA to substantially increase the ionic conductivity of a VE resin without decreasing $t(V = 0)$, unlike Trigonox 23A and CoNap, makes it a viable means for enabling the multiplex DC sensing of high-resistivity resins.

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4.2.2.2 Effects of Doping on the DC Sensing and Mechanical Properties of Manufactured Composite Panel. Figure 12 shows the SMARTweave voltage profile for a doped and nondoped VE panel, 12 × 12 square-inch 5 × 4 24-oz glass preform, during and after infusion. This figure shows that doping with TA greatly increases the S/N ratio of SMARTweave in a manufacturing environment, showing the viability of scale up. The increase in S/N ratio allows for faster cycle times and improved multiplex sensing of large parts with multiple nodes. In multiple node SMARTweave systems, where multiplexing cycle time is optimized, the noise in the system will increase significantly, necessitating the higher S/N ratio that can be achieved via doping.
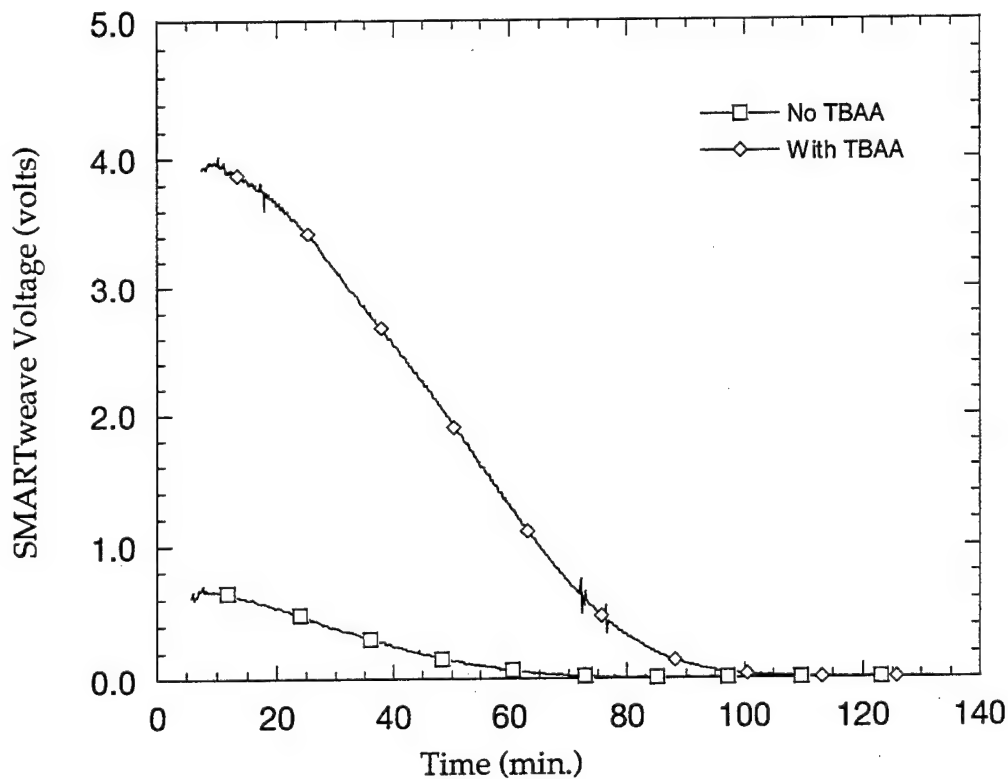
Figure 12. SMARTweave Voltage vs. Time in a SCRIMP Environment. Markers Do Not Represent All Data Points Taken. All Data Points Fall on Lines Connecting Markers.

Table 1 shows the results of the short-beam shear and open-hole compression tests. The panel that consisted of TA-doped VE resin showed a slight improvement in the average apparent

Table 1. Short-Beam Shear and Open-Hole Compression Test Results

Panel Samples	Apparent Compressive Strength (MPa)	Apparent Shear Strength (MPa)
Nondoped	219.2 ± 34.3	33.7 ± 0.7
TA-Doped	283.5 ± 39.8	40.9 ± 2.9

shear and compressive strength over that of the nondoped panel. The average apparent shear strength for the TA-doped specimen was 41 MPa, and 34 MPa for the nondoped specimens. The average apparent compressive strength of the TA-doped specimen was 284 MPa, and 219 MPa for the nondoped specimens. This shows that the addition of TA to VE resin not only affords a higher S/N ratio and decreases equipment cycle times but also does not adversely affect the mechanical properties of the part. Results in Table 1 show that doping may improve properties, but additional mechanical testing is required.

4.2.2.3 Determination of Dopant Ionization Constant and Equilibrium Concentration. Figure 13 shows the time it takes for TA-doped VE resin to reach ionic equilibrium concentration. As can be seen from Figure 13, 0.1-wt% TA in VE should be allowed to sit for at least 15 hr before being tested. However, it should be kept in mind that prepared samples are thoroughly mixed before shelving, which decreases the time needed for ionic equilibrium concentration to be reached.

Figure 14 shows the change of resistance with changes in the inverse concentration of ions. The inverse concentration of ions was determined with the use of equations (3) through (7), where the maximum theoretical molar ionic conductivity (Λ_{max}) for TA is 65.2×10^{-4} S-m^2/mol, and the formula weight (M_W) is 301.25 g/mol.

Figure 15 shows the concentration of ions vs. the weight-percent of TA added to VE. As the concentration of TA increases, so does the concentration of ions that ionize in solution with VE. According to these results, the trend is fairly linear and yields a fit with an intercept of 2.0×10^{19} ions and a slope of 2.4×10^{20} ions/m^3 wt%. With this known, one can tailor the conductivity of VE resin by simply adding in the appropriate amount of TA to yield the desired amount of ions.
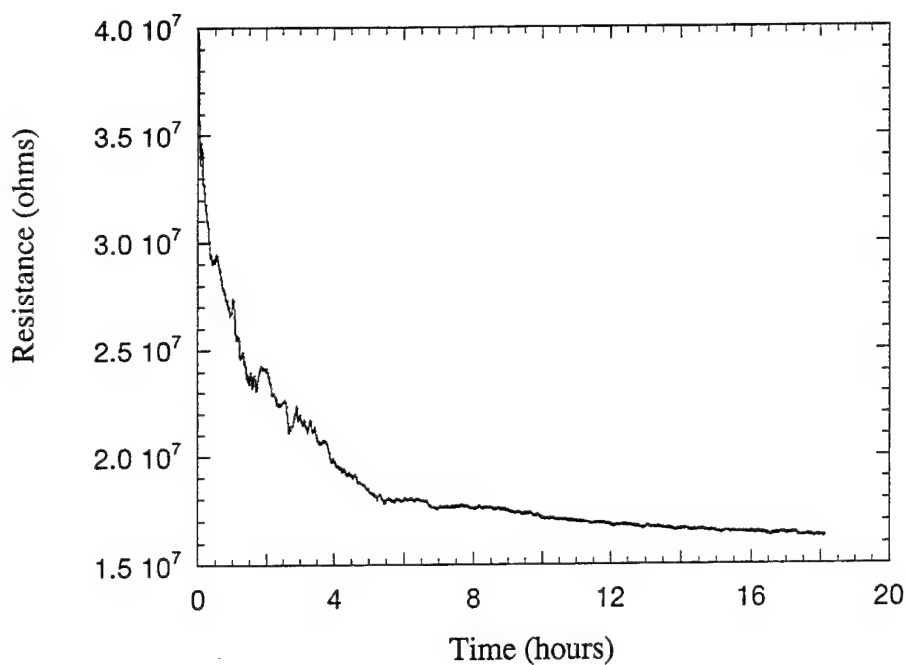
Figure 13. Resistance vs. Time for Equilibrium Concentration Study.
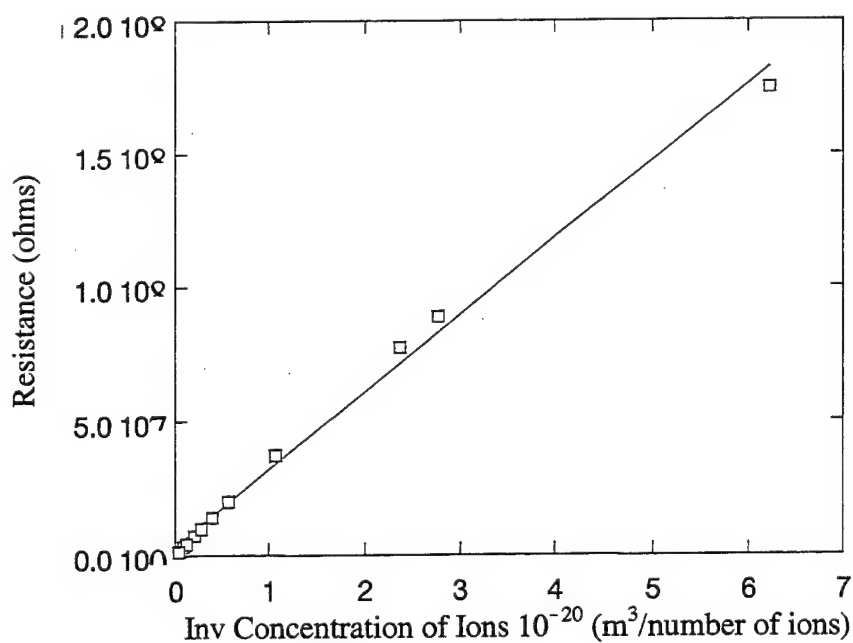
Figure 14. Conductivity vs. Inverse Concentration of Ions. Data Points Are Shown, Along With a Linear Curve Fit.
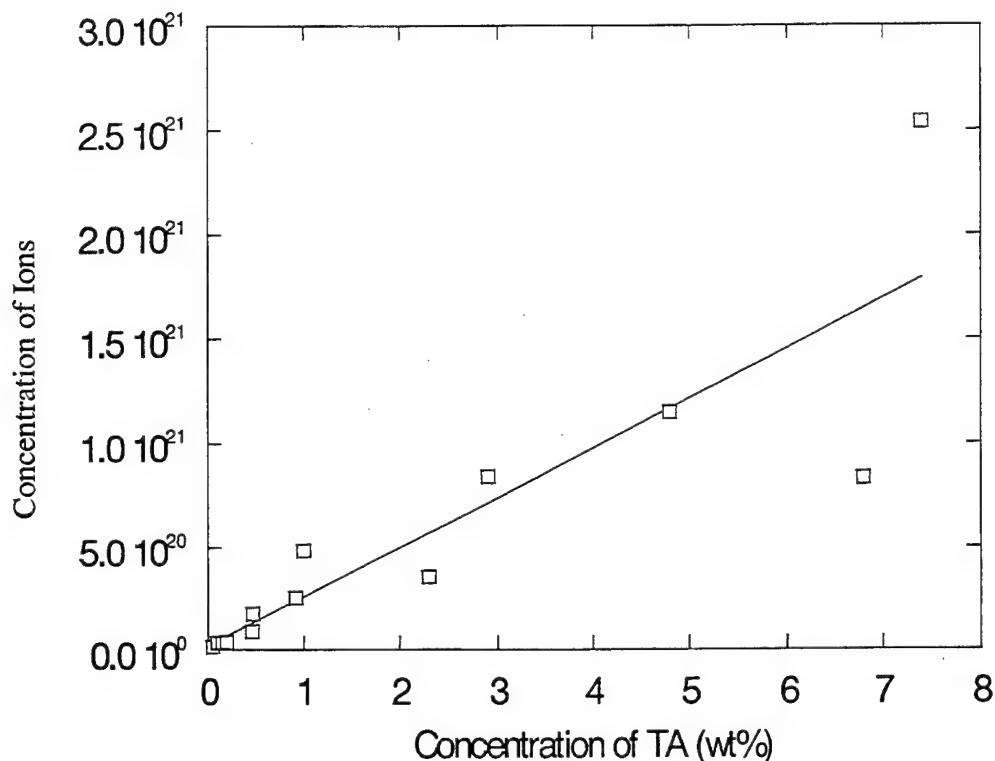
Figure 15. **Concentration of TA Ions vs. Mass Concentration of TA. Data Points Are Shown, Along With a Linear Curve Fit.**

4.2.2.4 Effects of TA on the Viscosity and Reaction Kinetics of Curing VE Resin. Figure 16 shows a comparison of a TA-doped and nondoped VE resin at 30 °C. As can be seen from Figure 16, TA seems to have an accelerating affect on the viscosity of curing VE resin at 30 °C. This may be the result of TA ions forming complexes with the ends of the VE monomer. Complex formation was also seen when sodium acetate was added to VE. In the case of TA, the formation of complexes may not be as extensive as it was in the case of sodium acetate, which would result in the ability of TA to raise conductivity without adversely affecting the mechanical properties of the cured resin system. However, the formation of complexes could cause the apparent viscosity to increase more rapidly than seen in the nondoped VE system.

Figures 1 through 19 show the degree of conversion of both the VE and ST monomers for the doped and nondoped systems at isothermal cure temperatures of 30, 40, and 50 °C.
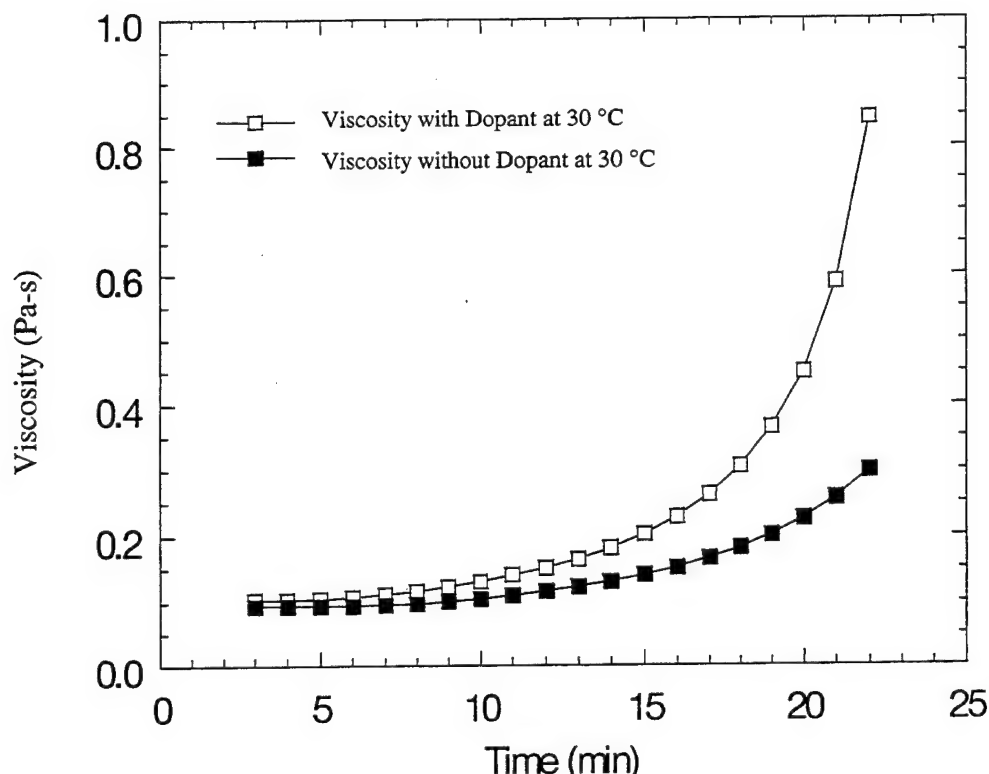
Figure 16. Effect of TA on Viscosity of Curing Doped 411-C-50 Resin. Data Points Shown Are With Lines.

Figures 17 through 19 also show that TA has no noticeable effects on the reaction kinetics of VE resin. This is consistent with the findings that the addition of TA to VE resin did not influence $V(t = 0)$. However, TA was found to increase the rate at which the viscosity of a curing VE resin increases. The period preceding the start of gelation is very short, less then 25 min at a cure temperature of 30 °C. The difference in times for the TA doped and nondoped resin to start gelling at 30 °C was less than 5 min. For T-FTIR tests at 30 °C, the time between measurements was 5 min. Therefore, it is completely possible that if TA does influence the reaction kinetics of VE resin before gel, these effects are small enough to go unnoticed in the experiments previously performed.
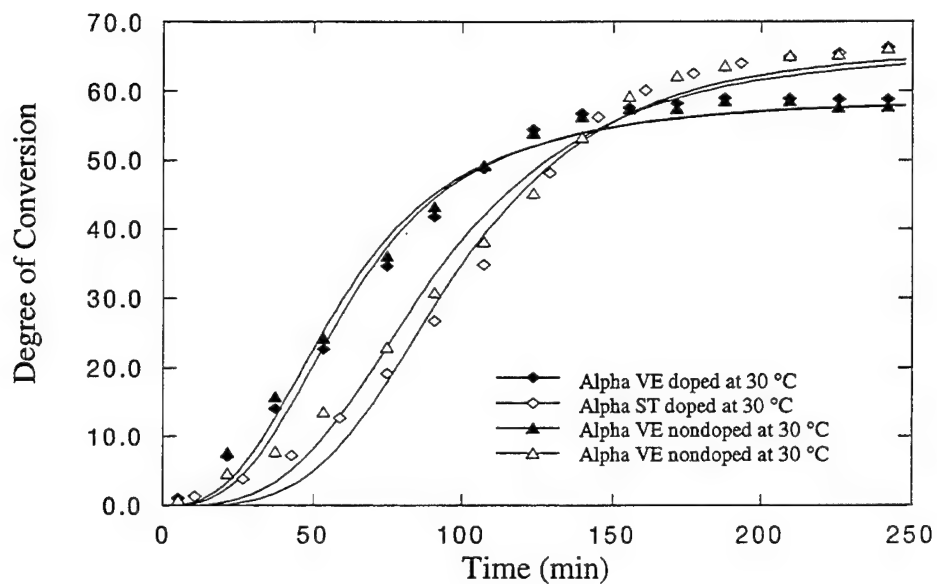
Figure 17. Degree of Conversion of VE and ST at 30 °C. Data Are Obtained Using T-FTIR. Markers Do Not Represent All Data Points Taken. Autocatalytic Curve Fit Is Shown as Solid Line.
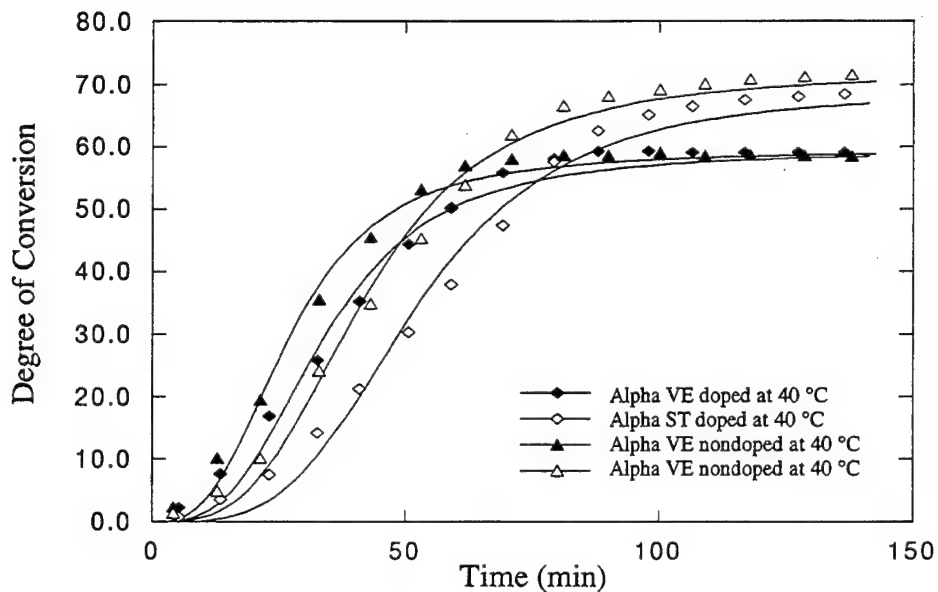
Figure 18. Degree of Conversion of VE and ST at 40 °C. Data Are Obtained Using T-FTIR. Markers Do Not Represent All Data Points Taken. Autocatalytic Curve Fit Is Shown as Solid Line.
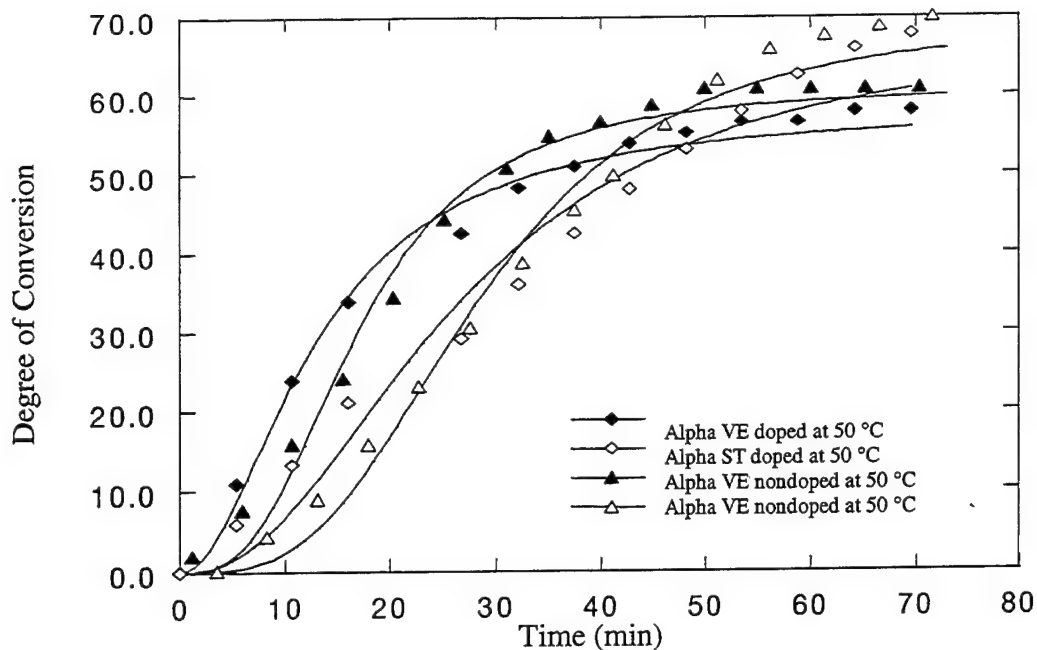
Figure 19. Degree of Conversion of VE and ST at 50 °C. Data Are Obtained Using T-FTIR. Markers Do Not Represent All Data Points Taken. Autocatalytic Curve Fit Is Shown as Solid Line.

5. Conclusions

The viability of using organic dopants to lower the resistivity of low-conductivity resins has been proven. TA and other tetrabutylammonium group ions have the ability to significantly lower the resistivity of VE resin without adversely affecting the cure behavior and mechanical properties of a manufactured composite part.

TA has the ability to increase the ionic conductivity of VE resins without shortening the duration of DC sensing. This investigation successfully characterized the ionization constant and concentration of TA in VE resin. TA was found to increase the rate at which the viscosity of a curing VE resin increases, without measurably affecting the degree of conversion of either VE or the ST monomers.

Another benefit of doping VE with TA is that it may enable the in-situ simultaneous measurement of ionic conductivity and degree of conversion of VE and ST monomers. This can be done through a modified SNTC, which can be embedded into a T-FTIR crystal. Through simultaneous measurements of degree of conversion and ionic conductance, the relationship between the electrical properties of a reacting polymer and reaction kinetics after gelation can be determined.

Doping VE resin with an organic dopant was proven as a viable method for lowering the resistance of high-resistivity resins. This investigation proved that doping enables the multiplex sensing of potentially thousands of point sensors using DC-sensing techniques such as SMARTweave.

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13. ABSTRACT (Maximum 200 words)

This investigation developed a methodology for doping high-resistivity vinyl-ester (VE) resins with an organic dopant. The polymeric resin system investigated was a Dow Derakane 411-C50 VE resin. A number of potential dopants were studied, and two in particular, tetrabutylammonium acetate (TA) and tetrabutylammonium iodide, were found to be capable of increasing the ionic conductivity of VE resin without adversely affecting the resin viscosity, mechanical properties, or reaction kinetics. The primary candidate dopant that was characterized in this investigation was a TA organic salt. TA, at a 0.1-weight-percent (wt%) concentration, was shown to have negligible effects on the mechanical properties and reaction kinetics of a curing VE part. TA was also found to slightly increase the rate at which viscosity increases, but not to the extent that would hinder resin transfer molding of a doped VE system. This investigation has proven that doping of VE resin with 0.1-wt% TA is a viable means of controlling and tailoring the conductivity of high-resistivity resins for the application of direct-current (DC)-sensing technology.

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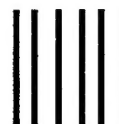**DEPARTMENT OF THE ARMY**

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